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(19) (CA) **CANADIAN PATENT** (12)

(54) Impregnating Medium for Cellulose-Containing Material

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# ABSTRACT

The present invention is concerned with an impregnating medium for cellulose-containing material, chiefly paper and wood. The active substance in this impregnating medium is a mixture of alkylsilanols and alkali aluminates which shows a large hydrophobising effect in only low concentrations. It has the advantage that it is employed as an aqueous solution so that it can also be employed within closed spaces and does not give rise to any problems with respect to ventilation of the spaces with moistening solvent. The solution is stable on storage; it contains preferably between 0.1 and 2.0 weight % alkylsilanol and between 0.03 and 0.1 weight % alkali aluminate.

The subject of the present invention is a new impregnating medium for cellulose-containing material, which makes this material water repelling. It contains as active substance, hydrolysis products of defined silanes, which, on mixing with an aluminate solution, show the desired effect.

It is known to employ alkylsilanes for rendering cellulose-containing materials hydrophobic. In addition, alcoholic solutions of comparatively large concentration which amounts for example to 10% and more are necessary; a really sufficient hydrophobising is not however achieved with such solutions. It is therefore also already known to add specific metal acid esters to the alcoholic silane solutions in order to improve in this way the hydrophobic effect. In this procedure, disadvantageously, it works out that the substances must be present in the alcoholic solution in comparatively great concentration in order to achieve sufficient results. Furthermore, the presence of alcohols as solvent increases the danger of undesired combustion and when working in closed spaces requires the installation of additional extraction apparatuses.

There therefore exists the task of finding a hydrophobising medium for cellulose-containing material, which exhibits its maximum effect in low concentrations and which is employed with a trouble-free solvent.

In fulfilment of this object, an impregnation medium for rendering cellulose-containing materials hydrophobic has been found which is characterised as aqueous or aqueous/alcoholic solution of a mixture of at least one alkali metal aluminate and at least one alkylsilanol (alkyl = ethyl, propyl or n-butyl). This mixture already shows a hydrophobising effect as 0.2% solution in water. Surprisingly, however, only the mixture of these two components possesses an impregnating effect, whereas both



the aqueous silanol solution and also an aluminate solution alone show no impregnating effect.

When rendering them hydrophobic, the cellulose-containing materials are immersed in the mixture according to the invention and then dried. It is however also possible to treat the material to be impregnated by brushing-on, spreading or spraying with the impregnating medium. It is however not necessary for the purpose of a good impregnation then to subject the treated material for a number of hours or days more to storage in a damp space as is recommended sometimes when employing alcoholic silane solutions. With the use of the impregnating and hydrophobising medium according to the invention, the material wetted with the solutions is rendered completely hydrophobic after the drying and can be used directly.

Cellulose in pure form as well as in its natural and synthetic modifications and working forms can be rendered hydrophobic with the aid of the impregnating medium according to the invention. By pure celluloses are to be understood for example paper, for example filter paper, or cellulosic fibres, whereas for the modifications or working forms are to be understood products such as pasteboard, cardboard, wood, chipboard, cotton fibres or sheets. The new impregnation material is suitably especially good for rendering hydrophobic paper in all its working forms, as well as also for the impregnation of wood with which likewise good results are achieved.

The alkylsilanols present in the solution employed according to the invention have, as alkyl groups, either ethyl, propyl or n-butyl groups. With other alkyl groups, stable aqueous or aqueous/alcoholic solutions are not obtained. The production of such solutions is described in German Patent

Specification .... (Patent Application P 30 37 220.4).

For details of this production, reference may be made to this protective right. In general, the preparation is achieved by dissolving the corresponding alkyltrialkoxysilanes in slightly acidified water and optionally then distilling off the alcohol forming in the hydrolysis. The alkoxy groups of these alkyltrialkoxysilanes are preferably those with 1 to 4 C atoms. The alkoxy groups of the alkyltrialkoxysilane are hydrolysed to a large extent in the aqueous or aqueous/  
10 alcohol solutions obtained in this hydrolysis and the alkylsilane is present in the solution predominantly as the alkyltrialkoxysilane. According to the invention, however, solutions may also be employed with which only one or two of the alkoxy groups of the alkyltrialkoxysilane were converted to hydroxyl groups.

A portion of the silanol group can also be present in partially condensed form with Si-O-Si groups, especially if the aqueous solutions have stood for a lengthy time. Not more than 10 Si-O-Si units should be present per molecule in  
20 this partial condensate. Such a partial condensation then occurs especially if concentrated solutions are employed as those preferably to be used.

The solutions according to the invention preferably contain between 0.1 and 2.0 weight % of alkylsilanol, especially preferably 0.3 to 1.0 weight %. The alkali metal aluminate is in general employed in amounts between 0.03 and 1.0 weight %, preferably between 0.1 and 0.4 weight %. Concentrated solutions can also be employed but in general no increase in the hydrophobising effect is achieved with such  
30 concentrated solutions.

The ratio by weight of alkylsilanol to alkali metal aluminate can fluctuate in wide limits between 1:10 and

10:1; preferably between 3.5 and 6 parts of alkali aluminate are employed to 10 parts of alkylsilanol.

Sodium and potassium aluminate are pre-eminently suitable for use as alkali aluminates; sodium aluminate is preferably employed.

The aqueous solution of the alkylsilanol can optionally even contain alcohol which arises from the hydrolysis of the corresponding alkyltrialkoxysilane. This alcohol does not need to be distilled off; as a rule. Its amount in the solutions preferentially to be employed lies at a maximum at 5 weight %, in general however between 1 and 3 weight %, related to the solution. With concentrated silanol solutions, the alcohol content of the impregnating medium according to the invention can also increase up to 10 weight %.

Example 1 Preparation of a silanol solution.

10 g of propyltrimethoxysilane are stirred at room temperature with 90 g of water to which 2 drops of 1% HCl are added, until after a few minutes a clear solution is obtained. The final solution contains in addition to the alcohol split off by hydrolysis, theoretically 7.4 g of propylsilanetriol.

This solution is hereinafter designated as solution a).

A commercially available 50% product which hereinafter is designated as solution b) is used as sodium aluminate solution.

Example 2 (First comparative Example)

25 g of solution a) are diluted with water to 100 g. Filter paper N°. 1375 of the firm Schleicher & Schull) in pieces sized 10 x 15 cm is immersed for 30 seconds in this solution, dried over night and then laid for drop testing on a glass plate. 0.5 cc of water are now applied by

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dropping. The drop is covered with a watch glass in order not to falsify the absorption time to be observed by air drying. In the present case, this precautionary measure is however unnecessary as the water drop is immediately soaked up by the paper.

The same results are obtained if 5 g or 50 g of solution a) are employed in the above manner.

Should soda Kraft paper having a weight of  $84 \text{ g/m}^2$  be used instead of the filter paper, rough side upwards, then  
10 the same results are obtained.

Example 3 (Second Comparative Example)

0.25 g of solution b) are diluted with water to 100 g. Filter paper as in Example 2 is immersed in this solution for 30 seconds, dried overnight and then laid for drop testing on a glass plate. 0.5 cc of water are now applied as a drop and evaluated as in Example 2. The water drop is immediately soaked up.

When the concentration of solution b) is doubled accordingly to 0.5 g in 100 g total solution, then the  
20 result remains unaltered. Only with further doubling to 1.0 g b) in 100 g total of solution is the water drop not absorbed for a few moments, but within 30 seconds.

Corresponding results are obtained if, instead of the filter paper, soda Kraft paper corresponding to that used in Example 2 is employed.

Example 4 Impregnation of paper.

5 g of solution a) are diluted with 94.5 g of water and 0.5 g of solution b) are added. Pieces of filter paper and soda Kraft paper according to Example 2 are dipped  
30 in this mixture, dried and tested with 0.5 cc of water as in Example 2. The water drop remains unchanged for many hours; in spite of covering up for about 50 hours, it is merely

evaporated. If the concentration of the mixture is reduced by half, corresponding to 2.5 g of solution a) and 0.25 g of solution b) in 100 g of impregnating solution, the same very good effect is achieved in the hydrophobising.

Example 5 Influence of sodium aluminate

Example 4, wherein 5 g of solution a) are employed is repeated, employing in 100 g of the impregnating solution varying amounts of solution b) corresponding to the following summary:

- 10 1. 0.5 g of solution b)
2. 0.25 g of solution b)
3. 0.125 g of solution b)
4. 0.06 g of solution b)

The water drop test corresponding to Example 2 indicates that with each of these solutions a very good hydrophobising effect was achieved. No water was soaked up by the paper and after about 50 hours, the water drop had merely evaporated.

Example 6 Hydrophobising of wood

20 15 x 15 cm large pieces of a phywood board 12 mm in thickness made of Limba wood are tested. The wood pieces are each dipped in the solution being tested and covered therewith within 1 minute. Then they are dried overnight and tested in the same way as the paper in the preceding Examples.

The results of the test at different concentrations are given in the following table.

	Concentration in 100 g of Impregnating solution	Result
30	2.5 solution a) + 0.25 g solution b)	Soaking up of the drop after about 1 hour
	5.0 g solution a) + 0.5 g solution b)	No soaking up of the drop



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Concentration in 100 g of Impregnating solution	Result
12.5 g solution a) + 1.25 g solution b)	No soaking up of the drop
1.25 g solution a) + 0.12 g solution a) (blank experi- ment).	Soaking up of the drop after about 1/2 hour.

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The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows :

1. Impregnation medium for rendering cellulose-containing material hydrophobic , characterized as aqueous or aqueous/alcoholic solution of a mixture of at least one alkali metal aluminate and at least one alkylsilanol , wherein alkyl stands for an ethyl , propyl or n-butyl group .

2. Medium according to claim 1, characterized in that the mixture consists of said alkali metal aluminate and said alkylsilanol in solution in the ratio 1:10 to 10:1 .

3. Medium according to claim 1 or 2 , characterized in that it contains said alkali metal aluminate in amounts between 0.03 and 1.0 weight percent and said alkylsilanol in amounts between 0.1 and 2.0 weight percent .

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